Disclaimer:
This English translation is produced by machine translation and may contain errors.The JPO, the INPIT, and and those who drafted this document in the original language are not responsible for the result of the translation.

Notes:
1. Untranslatable words are replaced with asterisks (****).
2. Texts in the figures are not translated and shown as it is.

Translated: 07:42:36 JST 05/01/2008
Dictionary: Last updated 04/11/2008 / Priority: 1. Chemistry / 2. Natural sciences / 3. Medical/Pharmaceutical sciences

FULL CONTENTS

[Claim(s)]
[Claim 1] They are a kind of an organic acid, or two sorts or more 0.01-5.0 The extract extracted from gluten which is wheat protein in the 1-20-volume % ethanol aqueous solution of which weight / capacity % content is done is used as the poly cation wall membrane material. The manufacture method of the microcapsule characterized by making the poly anion wall membrane material and a complex coacervation cause.
[Claim 2] The manufacture method of a microcapsule according to claim 1 that an organic acid is a kind of citric acid, lactic acid, malic acid, and acetic acid, or two sorts or more.
[Claim 3] The manufacture method of a microcapsule according to claim 1 or 2 that the poly anion wall membrane material is a kind of gum arabic, sodium alginate, and agar, or two sorts or more.
[Claim 4] Claim 1 - three microcapsules obtained by a method given in any 1 clause.

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the manufacture method of a microcapsule.
[0002]
[Description of the Prior Art] As the manufacture method of a microcapsule, many things are known conventionally, and there is the complex coacervation method as one of them. The complex coacervation method mixes two sorts of aqueous solutions, poly cation colloid and poly anion colloid. It is the method of making phase separation occurring by an electric interaction, producing two, a thick colloid phase and a thin colloid phase, and using a thick colloid phase as wall membrane of a capsule. That is, it is the method of distributing the thing used as a heart substance in both colloid, making the complex of wall membrane material forming in the surroundings of a heart substance at the time of the phase separation by an electric interaction, and making a heart substance wrapping in and encapsulating. For example, it is encapsulating by making an electric interaction start by pH movement, using a gum arabic aqueous solution as poly anion colloid, using a gelatin aqueous solution as poly cation colloid. The protein which has zwitter ion, such as albumen, casein, and collagen, in addition to the above-mentioned gelatin as a poly cation wall membrane material used for the complex coacervation method is known. It is dissolving in inexpensive solvents, such as water, as an absolute necessary condition of such

poly cation wall membrane material. However, while the gluten which occupies the subject of comparatively inexpensive wheat protein also in protein with the same zwitter ion is insoluble in water, viscosity is not dramatically used as wall membrane material polymer strongly until now.
[0003] Gluten is classifiable into gliadin of solubility, and insoluble glutenin with a 50-70-volume % ethanol aqueous solution. Although the viscosity of gliadin of the solution is low in a 50-70 volume % ethanol aqueous solution and the conditions of the poly cation wall membrane material are prepared Natural gums, such as gum arabic which is the well-known poly anion wall membrane material, caused precipitation or coagulation momentarily in the 50-70 volume % ethanol aqueous solution, and encapsulation was impossible by the complex coacervation method in the combination of these two wall membrane material. Although the method (JP,H2-138951,A, Japanese Patent Application No. No. 309715 [63 to]) of using only gliadin and on the other hand manufacturing a microcapsule with an emulsification spray drying process etc. is proposed Are inferior to the long-term stability of the pulverization thing of the substance which does not have oxidation stability since the complex coacervation method is not used fundamentally. Moreover, since it is necessary to operate it in high-concentration ethanol in order to obtain stabilization powder enough by this manufacture method, there are problems, like there is the necessity of using the recovery subsystem and all the equipment of a solvent as a gas proof apparatus. This invention offers the manufacture method of a new microcapsule of having used the unused wheat protein while solving the above problem.
[0004]
[Means for solving problem] [this invention person etc.] by using the solution in which the aqueous solution of low ethanol concentration was made to dissolve an organic acid as an extracting solvent of gluten, as a result of inquiring wholeheartedly in view of the above-mentioned point It found out that it was available considering the solution which dissolved the extract of the gliadin subject extracted, or its dry matter as poly cation wall membrane material colloid. Furthermore, rather than the gliadin emulsification spray drying process (JP,H2-138951,A, Japanese Patent Application No. No. 309715 [63 to]) which existed conventionally, this invention method also finds out that it is the method of making the outstanding thing oxidation stability of the substance in which oxidation stability is inferior, and came to complete this invention. That is, this inventions are a kind of an organic acid, or two sorts or more 0.01-5.0 The extract extracted from gluten which is wheat protein in the 1-20-volume % ethanol aqueous solution of which weight / capacity % content is done is used as the poly cation wall membrane material. It is related with the microcapsule obtained by the manufacture method of the microcapsule characterized by making the poly anion wall membrane material and a complex coacervation cause, and this method.
[0005] the extracting solvent used by this invention -- a 1-20 volume % ethanol aqueous solution -- desirable -- a 5-10 volume % ethanol aqueous solution -- 0.01-5.0 the organic acid of weight / capacity % -- desirable -- 0.2-0.5 The organic acid of weight / capacity % is dissolved. as an organic acid -- for example, a kind of citric acid, lactic acid, malic acid, and acetic acid -- or two or more sorts of especially citric acid is used preferably.
[0006] Although any of the gluten (raw gluten) separated by the conventional method or its dry powder (vital gluten) are sufficient as the wheat protein used by this invention, it is desirable to use the easy dry powder of handling. Extraction operation of 1 to 3 hours is performed keeping solution temperature at 20-30 degrees C by attached extraction tubs, such as a propeller, using this 7-10-times the amount extracting solvent of powder gluten as an extraction condition. Then, operation of centrifugal separation

operation or filtration operation separates into settlings (insoluble thing) and supernatant liquor (soluble thing), and supernatant liquor is used as it is. Or you may use the solution used as the after-desiccation aqueous solution. Moreover, what is necessary is just to determine about the separation degree of supernatant liquor with the dissolution amount of resources of the 70-volume % ethanol aqueous solution of the evaporation-to-dryness thing of supernatant liquor. That is, if the 70-volume % ethanol aqueous solution dissolution thing is contained 70weight % or more in the evaporation-to-dryness thing, it can be used as a poly cation wall membrane material in this invention method. However, even if it is 70 or less weight %, it cannot be overemphasized that it is usable depending on the purpose.

[0007] In addition, even if it performs microencapsulation of an unstable heart substance to oxidation using the water-soluble protein (wheat albumin, globulin, etc.) contained in wheat protein with the same manufacturing method as this invention, unlike the case where gluten is used, a microcapsule stable enough cannot be obtained.

[0008] It is not limited especially as a poly anion wall membrane material used by this invention, for example, a kind of well-known material, such as gum arabic, sodium alginate, and agar, or two sorts or more are used. It is gum arabic which excelled [low viscosity] in solubility preferably. As for the concentration in colloid of wall membrane material, about 1-10 percentages by weight are good, the amount of 1 / 1 - 1/10 used of the poly cation wall membrane material is suitable for the poly anion wall membrane material, and 1 / 7 - 1/10 are preferably good.

[0009] As an object of the heart substance of this invention method, the solid powder of water-insolubility or lipophilicity vitamin (vitamin A, D, E), a lipophilicity flavor, a pigment, a lipid, etc. are mentioned. [the operation method of the complex coacervation of this invention method] [the aqueous solution which dissolved the extract (4 to 9 weight % of solid concentration) or extraction solid matter extracted by this invention method as poly cation colloid so that it might become 4 to 9weight %] pH of a solution using an organic acid 3.5-4.0 It adjusts so that it may become. It is made to descend until it adds 1 (the heart amount of substance which corresponds to 20 to 30weight % in the amount of total solids in a final product is applied) to 10weight % of poly anion colloid and pH is set to about 3 with organic acids, after putting a heart substance into this solution and making it emulsify or distribute, and a complex coacervation is made to cause. The solution which caused the complex coacervation can perform pulverization by desiccation operation of spray drying etc. after direct or concentration, when it does not need hardening of a microcapsule film. Moreover, when there is the necessity for hardening, it is possible to harden with common knowledge curing agents, such as alum, tannin, and formalin. By this invention method, the pulverization thing (microcapsule) of the substance with which vitamin A etc. oxidizes easily demonstrates the oxidation stability which was excellent even if exposed into the air for a long period of time. moreover, the solution of this substance hardly causes membranous destruction under low pH (2-4) and high temperature heating (80-120 **) -- it excels.

[0010]

[Working example] Although a work example is given and this invention is explained still in detail hereafter, this invention is not limited to these.

[0011] It is 5g of citric acid to 1500ml of ethanol aqueous solutions of a work example 1 and 110 volume % of comparative examples. It dissolved, powder gluten (80 weight % of protein, 10 weight % of moisture, lipid 1.2 weight % and ash content 1.0 weight %) 200g was put in, and extraction was performed for 2 hours. The extract dissociated on 4000rpm and the conditions for 30 minutes with the desk-type centrifugal separator (100ml x4 **), and used supernatant liquor as poly anion colloid as it

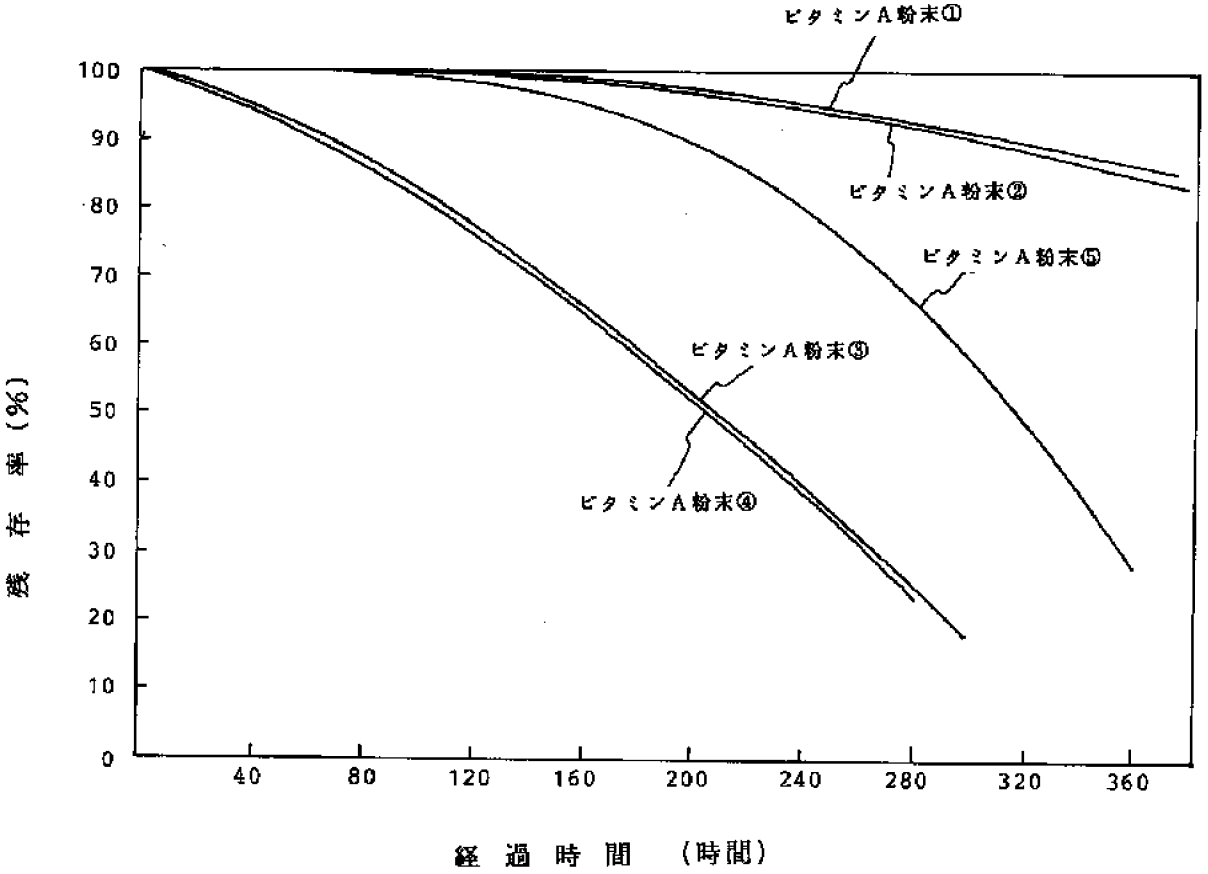
was (5 weight % of solid concentration). Subsequently, 200g of supernatant liquor is warmed at 70 degrees C, 0.5g of citric acid is put in into it, and it is pH 3.7 It carried out. The vitamin A palmitate (170 10,000 IU/g) 4.5g which carried out dissolution fusion of 1weight % of the monoglyceride was added to this solution, and high-speed emulsification between dichotomy was performed by homomixer.
[0012] Then, 200g of gum arabic aqueous solutions were added 1weight %, 3g of citric acid was put in with low-speed rotation, and churning was continued for 5 to 10 minutes. This solution was used as powder using the spray dryer (vitamin A powder **). Moreover, what dried some above-mentioned supernatant liquor and was used as powder is dissolved with water so that it may become concentration 5%, 0.5g of citric acid is put in, and it is pH 3.7. The powder of vitamin A was obtained by the same operation as the above using the solution carried out (vitamin A powder **).
[0013] Next, as following, it carried out and the comparison article was manufactured. Powder gluten 10g 1g of citric acid It distributed to the 10% ethanol 200g which dissolved, monoglyceride content vitamin A palmitate 4.5g was added 1%, and the powder of vitamin A was obtained like the above (vitamin A powder **). wheat water-soluble protein (82 weight % of protein, albumin, and a globulin subject --) 6.0 weight % of moisture, and lipid 8.0 Weight % and ash content 4.0 Weight %10g Gum arabic 2g dissolving in 400ml of water -- warming -- monoglyceride content vitamin A palmitate 4.5g was added after 1%, and the powder of vitamin A was obtained like the above (vitamin A powder **). The 70-volume % ethanol aqueous solution extracted gliadin from gluten, and vitamin A powder was obtained by the method (emulsification spray drying process) of the range octavus clause (a work example 1, comparative example 1) of the application for patent of JP,H2-138951,A (vitamin A powder **). Vitamin A powder ** - **, vitamin A powder ** - ** (comparative example) were saved in 50-degree C oven, the rise and fall of vitamin A were temporally made a fixed quantity, the amount of vitamin A before a retention test was made into 100 %, and aging of residual ratio was measured. A result is shown in drawing 1 . It was checked that the vitamin A powder obtained by this invention method from this result is far more stable than the vitamin A powder obtained from the conventional method or other protein in wheat.
[0014] An aqueous solution which becomes 10 weight % about vitamin A powder ** made as an experiment in the work example 2 and the comparative example 2 work example 1 - **, vitamin A powder [of a comparative example 1] ** - ** is made, citric acid is added, and pH of a solution is 3.0**0.2. It adjusted so that it might become. What poured these 10ml of solutions distributively in the test tube Heating was examined at 120 degrees C for 5 minutes, and thermal stability was examined with the monograph affair of heating and 50-degree C 60-day preservation at 80 degrees C for 60 minutes. A test result is shown in Table 1. It was checked that the microcapsule obtained by this invention method from this result has thermal stability farther than the microcapsule obtained from a conventional method or other wheat protein.
[0015]
[Table 1]

ビタミンA粉末	①	②	③	④	⑤
120℃　5分	分離なし	分離なし	完全分離する	完全分離する	多少分離を起こす
80℃　60分	分離なし	分離なし	分離を起こす	完全分離する	殆ど分離を起こさない
50℃　60日	分離なし	分離なし	2日で分離が始まる	1日で分離が始まる	35日頃より分離が始まる

[0016]
[Effect of the Invention] As explained above, in the former, this invention used gluten in the wheat protein which was not made, and became possible [manufacturing the microcapsule by the coacervation method]. Moreover, since gluten can be used, while it is inexpensive and manufacture of a microcapsule with quantitative stability is enabled Enabling mass production by low cost, the microcapsule by this invention method made possible further the microcapsule which an uncured thing can also bear also at high temperature heating (80-120 **: 60 minute - 5 minutes) under low pH (pH:2-3) and low pH. Since all the raw materials consist of a food material or a food additive except for some curing agents used by request, use with edible [wide range from the first] is possible for the microcapsule raw material by this invention method.

[Brief Description of the Drawings]
[Drawing 1] Drawing 1 is a graph which shows the heating stability of the vitamin A powder of a work example 1 and a comparative example 1.

[Drawing 1]



[Translation done.]